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Summary of Comments and Responses for Methods 5B and 5F

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Research Triangle Park, NC

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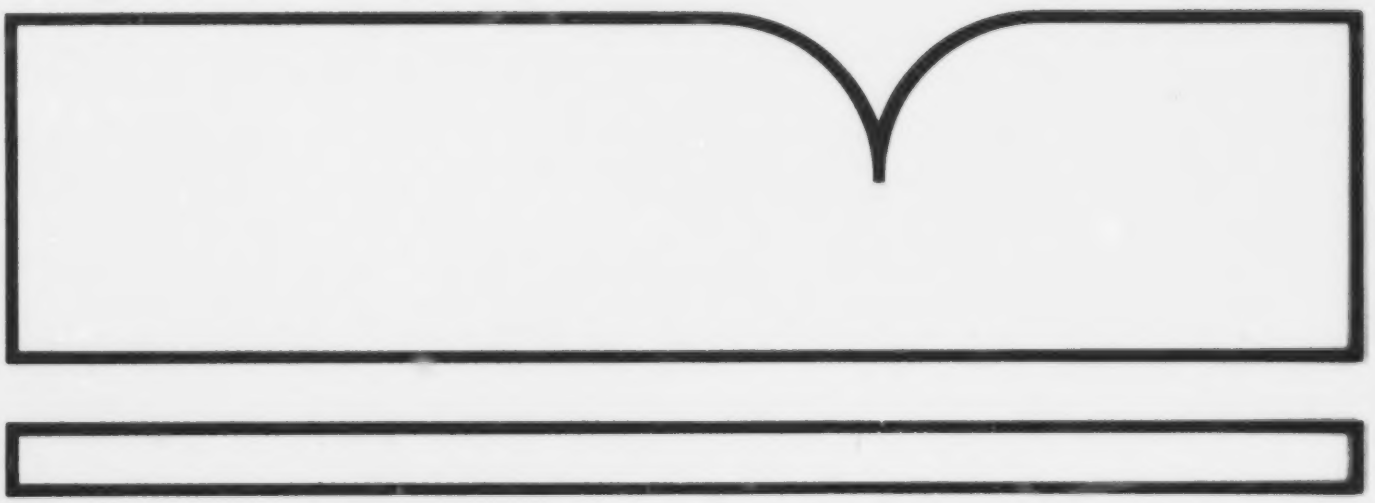

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Office of Air Quality
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Research Triangle Park NC 27711

EPA-450/3-86-008
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Air

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Two new methods for measuring particulate matter from stationary sources without measuring condensed sulfuric acid have been developed. The public comments on the proposed methods have been summarized along with the Agency's responses to the comments

17. KEY WORDS AND DOCUMENT ANALYSIS

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Emission Standards and Engineering Division

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Air and Radiation
Office of Air Quality Planning and Standards
Research Triangle Park, NC 27711

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LIST OF ACRONYMS USED

EPA - Environmental Protection Agency.

FCCU- Fluid catalytic cracking unit.

FGD - Flue gas desulfurization.

IPA - Isopropyl alcohol.

NSPS- New source performance standards

SIP - State Implementation Plan.

Chapter 1

INTRODUCTION

On May 29, 1985, the U.S. Environmental Protection Agency (EPA) published in the Federal Register (50 FR 21863) Method 5B, "Determination of Nonsulfuric Acid Particulate Matter from Stationary Sources," and Method 5F, "Determination of Nonsulfate Particulate Matter from Stationary Sources." These methods were proposed under the authority of Sections 111, 114, and 301(a) of the Clean Air Act, as amended.

Public comments were solicited at the time of proposal. To provide interested persons the opportunity of oral presentation of data, views, or arguments concerning the proposed revisions and test methods, a public hearing was scheduled for July 16, 1985, at the Research Triangle Park, North Carolina, but no person desired to make an oral presentation. The public comment period was from May 19, 1985, to July 31, 1985.

Nine comment letters were received concerning issues relative to the proposed test methods. A detailed discussion of these comments and responses is summarized in this document. The comments have been carefully considered, and necessary changes have been made in the proposed methods.

Chapter 2

SUMMARY OF CHANGES SINCE PROPOSAL

The following changes have been made in the rule since proposal.

1. Section 4.3, Method 5F. Section 4.3.3 has been revised to change the beaker size from 600 ml to 250 ml.

2. Section 4.4, Method 5F. Section 4.4.1 has been added to include a filter blank in the analysis procedure.

3. Section 4.4, Method 5F. Section 4.4.2 has been added to describe the treatment of the water blank.

Chapter 3

SUMMARY OF PUBLIC COMMENTS AND RESPONSES

Commenter IV-D-1, IV-D-3

1.1 Comment: Inasmuch as the Environmental Protection Agency's (EPA's) intention was not to include sulfuric acid (H_2SO_4) when the emission standards for particulate matter were established for facilities subject to Subparts D, Da, Db, and J, these amendments will provide more meaningful measurements of particulate emissions.

Response: No response is necessary.

Commenter IV-D-2

2.1 Comment: In the "Supplementary Information" section of the proposal, the recognition that the proposed methods "may not be appropriate" for emission limits that are based on achieving a particular impact on ambient air quality should be changed to "are not appropriate." Otherwise, considerable pressure will be placed on control agencies to allow these methods on existing sources.

Response: The phrase "are not appropriate," was not used because it would prevent State governments from choosing Methods 5B and 5F, when they are deemed appropriate to their programs. However, we believe that Methods 5B and 5F would not be appropriate in the majority of these cases. The paragraph cited in the comment has been revised to clarify its meaning.

2.2 Comment: The report, "Impact of Temperature and Filter Material on Combustion Source Particulate Results," did not conclusively prove that particulate weight reductions achieved by sampling at elevated probe/filter temperatures and subsequent drying of samples at elevated temperatures were due to avoiding H_2SO_4 collection and vaporizing H_2SO_4

after collection. The fact that extraction of samples with isopropyl alcohol (IPA) did not yield weights equivalent to weight reductions on heating of the samples may mean that particulate matter other than H_2SO_4 is being lost through these processes.

Response: We agree that this report does not present conclusive proof that sampling and subsequent drying at elevated temperatures eliminate only H_2SO_4 from particulate matter samples. However, two other reports in the docket present more convincing evidence. These two reports are "Evaluation of the Particulate Test Method for Fluid Catalytic Cracking Unit (FCCU) Regenerator Emissions" and "Evaluation of Method 5B at a Coal-fired Boiler." As discussed in these reports, paired samples from both FCCU's and fossil-fuel fired boilers showed good agreement when one sample was measured by the water extraction procedure (Method 5F) and the other sample was heated to 160°C before being extracted. If material other than H_2SO_4 was lost during the heating step, then the particulate matter concentrations from the latter analysis would have been significantly lower than those from the former analysis. Because this was not the case, we concluded that the heating step removed only H_2SO_4.

2.3 Comment: The primary intent of these amendments is to minimize the measurement of H_2SO_4 mist on particulate samples. We believe that the proposal has not succeeded in accurately eliminating that specific portion of the sample. As indicated in Comment Number 2.2, heating of the sample as dictated by Method 5B may eliminate particulate matter other than H_2SO_4. Method 5F, on the other hand, permits the exclusion of all water soluble sulfates.

Response: As discussed in the response to Comment 2.2, we believe that Method 5B does not eliminate particulate matter other than H_2SO_4. Method 5F does exclude water soluble sulfates. However, its use is limited to FCCU's that are not equipped with wet flue gas desulfurization (FGD) units. We believe that any water soluble sulfates at these facilities would be limited to condensed H_2SO_4 or metal sulfates formed from condensed H_2SO_4 in the sampling train. Thus both methods minimize the measurement of H_2SO_4 only.

Commenter IV-D-4

4.1 Comment: The same rationale which EPA has applied to fossil fuel-fired generators and petroleum refineries should also be applied to other sources which emit sulfur oxides and particulate matter. We have shown that gaseous compounds in the offgases of copper smelters can combine to form particulate matter within and after the control equipment. Thus primary copper smelters are subject to the same conditions which prompted EPA to propose a method to accommodate the utility and petroleum refinery industries. In conclusion, EPA should adopt similar amendments to test methods for primary copper smelters and other similar sources.

Response: Our action in proposing Methods 5B and 5F to measure particulate matter without measuring condensed H_2SO_4 was a specific response to a specific problem. When the particulate matter emission standards were established for the industries affected by this proposal, the feed stocks in use were relatively low in sulfur content. Consequently, emissions of sulfur oxides were relatively low, and the potential for collecting condensed H_2SO_4 along with the particulate matter sample was also low.

Because the data base to support the emission limit for both of these industries were collected using Method 5, any potential for measuring condensed H_2SO_4 was considered in establishing the emission limit.

After the particulate matter standard for these industries had been established, new control techniques for sulfur dioxide (SO_2) emissions allowed the use of higher sulfur content feed stocks. Although SO_2 controls reduce SO_2 emissions, they have little effect on sulfur trioxide (SO_3) emissions. Conventional particulate matter control devices are also unable to reduce SO_3 emission. Thus, the net effect of using higher sulfur feed stocks is an increase in the level of SO_3 in the gas stream. Under certain conditions, this SO_3 condenses to form H_2SO_4 and is collected in a Method 5 sampling train as particulate matter.

Because little or no condensed H_2SO_4 was measured with the particulate matter when the emission limit was being established, then H_2SO_4 should not be measured with the particulate matter when determining compliance with this emission limit. This is the specific problem that Methods 5B and 5F were designed to address. The issue is not simply that Method 5 may measure condensed H_2SO_4 as particulate matter. Rather, the issue is that the potential for condensed H_2SO_4 has increased greatly because the sulfur content of the feed stocks now being used is much higher. There is no evidence that a similar situation exists for copper smelters.

4.2 Comment: We applaud the realization by EPA that H_2SO_4 mist is indeed difficult to control, especially when it is generated by the control equipment. It is also laudable that EPA has decided to propose changes to Method 5 to accommodate this problem. However, we believe that EPA must

investigate methods other than the simple increase in filter temperature to minimize H_2SO_4 collection on the filter.

Specifically, we believe that the following factors affect the adequacy of the proposed test methods.

1. The H_2SO_4 normally exists with eight water molecules of hydration instead of two as stated in the EPA proposal.

2. When glass fiber filters are used to collect particulate matter samples, the amount of condensed H_2SO_4 collected varies directly with the size of the filter.

3. While maintaining a sampling temperature greater than 160°C (320°F) can be difficult, it is not an insurmountable problem. We have shown in these preceding comments that merely increasing the filtration temperature is inadequate and continues to penalize sources with high acid dew points.

Response: We believe that we have demonstrated for sources covered by this proposal that increasing filtration temperature accompanied either by additional heating after collection (Method 5B) or by water extraction with subsequent analysis for sulfate (Method 5F) will eliminate the condensed H_2SO_4 contribution to particulate matter measurements. Thus, questions about the number of water molecules associated with H_2SO_4 at equilibrium is not an issue with either of these methods. In Method 5B, any H_2SO_4 collected in sampling is driven off by heating in the laboratory while in Method 5F any collected H_2SO_4 is converted to the nonhygroscopic ammonium sulfate before weighing.

We agree that, although it is difficult, it is possible to maintain sampling temperatures above 160°C (320°F). However, for the sources

covered under this proposal we do not believe that there is a need to maintain sampling temperatures above this level.

The data submitted by this commenter do show a relationship between filter size and H_2SO_4 collection. This is most probably related to a reaction between the H_2SO_4 and the filter media. Methods 5, 5B, and 5F specify that glass fiber filters used at sources containing SO_2 and SO_3 must be of a type that are unreactive to these gases. Guidance on the selection of such filter materials can be found in the report "Inertial Cascade Impactor Substrate Media for Flue Gas Sampling." Copies of this report are available for a fee from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

4.3 Comment: In 40 CFR Part 51, Appendix B, EPA promulgated a particulate standard based on the State of Colorado process weight curve for sources with reasonably available control technology. This curve was formulated in the same manner as the Bay Area Air Pollution Control District process weight regulation developed in 1959. The curve was determined by testing the best controlled sources in the Bay Area and fitting the results under a curve. The test method used was similar to the American Society of Mechanical Engineers Power Test Code method which uses an in-stack filter for determination of particulate. Obviously, the method would measure particulate at control device temperatures and "condensible material present at the gas phase" would not be measured. A complete discussion of this matter was formally submitted to the EPA on September 25, 1979, by the Phelps Dodge Corporation. Thus, the standard for primary copper smelters is based on a test method which did not include condensible material. In the May 29, 1985, Federal Register, EPA states

that it "undertook a study to develop a method consistent with the original standards" promulgated for fossil fuel fired power plants and FCCU's. With regard to primary copper smelters, EPA adopted a standard which was inconsistent with the test method mandated for their use. Phelps Dodge now seeks to be treated in a manner similar to that afforded the aforementioned sources to have a test method compatible with control equipment installed in compliance with State and Federal regulations. We, therefore, believe that EPA should, either change the particulate standard for copper smelters based on a study using Method 5 or apply Method 5B to copper smelters as well as to the industries cited in the May 29, 1985, Federal Register.

Response: This comment is not applicable to this proposed action. The regulation referenced was adopted under the State Implementation Plan (SIP) program. Methods 5B and 5F are proposed for use only at sources regulated under the new source performance standards (NSPS) program. Actions taken under this program have no direct effect on the SIP program. Any comment on regulations adopted under the SIP program should be made directly to the State in which the regulation was adopted.

Commenter IV-D-5

5.1 Comment: The present particulate standard for Subpart D and Da units with wet scrubbers was based upon test data obtained before wet scrubbers. Therefore, to change the testing location to after the wet FGD scrubber without positive data to show no increase in particulates due to the scrubber is in fact changing the standard.

Response: The original intent of this regulation was to require testing downstream of any FGD unit. Because of concern that condensed sulfuric acid following the FGD would be counted as particulate matter by

Method 5, sources were temporarily given the option of testing upstream of the FGD. Method 5B will not measure the condensed sulfuric acid and, therefore, may be used downstream of the FGD unit as originally intended by the regulation. The change in test location does not represent a change in the standard, but merely eliminates a temporary option granted until a method could be developed that would not count condensed sulfuric acid as particulate matter.

In addition, during its development Method 5B was shown to yield particulate matter measurements that were always equal to or less than those measured by Method 5 at the same location. Since the promulgation of Subpart Da, at least three sources equipped with FGD units have conducted compliance tests where the test point was downstream of the FGD unit and the particulate matter was measured using Method 5. All of these sources were found to be in compliance with the particulate matter standard. Because the particulate matter measured by Method 5B is always equal to or less than that measured by Method 5, these sources would also have been shown to be in compliance if Method 5B had been used to measure the particulate matter. Therefore, we believe it is reasonable to require that sources equipped with FGD units must conduct particulate matter tests downstream of the FGD unit.

Commenter IV-D-6

6.1 Comment: See Commenter IV-D-1, Comment 1.1.

Commenter IV-D-7

7.1 Comment: Method 5B is designed to minimize the measurement of H_2SO_4 mist as particulate matter from boilers employing a wet FGD system. The rationale for doing so is that under conditions of high moisture content and with SO_3 present in the flue gas, the acid dew point occurs at

temperatures above the allowed Method 5 collection temperature. This allows SO_3 to condense on the filter thereby increasing the measured particulate mass.

We believe it would be appropriate for EPA also to allow the use of Method 5B on combination wood/oil and wood/coal boilers subject to NSPS, whether equipped with scrubbers or not, and even if the scrubbers are not used for FGD. Similar flue gas conditions, including high moisture content, can occur with these boilers which leads to the condensation of H_2SO_4 mist and collection as particulate matter by Method 5.

We have substantial data which indicate particulate catches from such combination boilers can contain 10 to 40 percent soluble sulfates when Method 5 is used to determine SIP compliance.

Response: See response to Comment 4.1.

Commenter IV-D-8

8.1 Comment: The approach and terminology used for Method 5F are confusing since it appears that all water soluble material (including H_2SO_4) is being removed from the particulate matter determination. If the intent was only to remove H_2SO_4, then why was a water extraction procedure used? Water will remove any water soluble material whereas an IPA extraction would only remove H_2SO_4. Why should water soluble material (other than H_2SO_4 mist) be removed from the particulate matter determination?

Response: Though Method 5F does include a water extraction as part of its analysis procedure, an aliquot of this extract is subsequently analyzed for sulfate content while the remainder of the sample is dried and weighed. Only the measured sulfate content is subtracted from the final particulate matter weight. Thus, not all water soluble material is

eliminated from the particulate matter determination; only the water soluble sulfates are eliminated. Though there are water soluble sulfates other than H_2SO_4, we believe that the only source of water soluble sulfates at the facilities for which we propose the use of Method 5F is H_2SO_4. Thus, eliminating water soluble sulfates from the sample eliminates only H_2SO_4.

We agree that an IPA extraction would remove only H_2SO_4 and not other sulfate compounds. However, our experimental results indicated that IPA was not always 100% effective in extracting H_2SO_4. For these reasons, we believe that the water extraction procedure proposed in Method 5F is appropriate for eliminating H_2SO_4 from particulate matter samples.

8.2 Comment: It seems to me that EPA needs to review the data of other researchers in more detail before eliminating the use of higher temperatures for the sample probe and 100 percent IPA extraction to remove H_2SO_4 from particulate matter. The EPA Environmental Sciences Research Laboratory researchers (EPA 600/3-84-056) have developed a miniature acid condensation system and stated in their report that "a procedure can be followed to separate absorbed H_2SO_4 from metallic sulfates. The procedure involves washing the filter, the filter holder, and the probe through an SO_4-free filter media using 100 percent IPA. This recovers the H_2SO_4 alone. Following this, the particulates can be washed with 80 percent IPA/20 percent H_2O_2 to recover the remaining water soluble sulfates separately." Note that their probe and filter temperature is 271°C (520°F). The EPA Industrial Environmental Research Laboratory (EPA 600/7-79-156,159) recommended 316°C (600°F) for the probe and 281°C (550°F) for the filter. Other researchers outside EPA have come to similar conclusions. Brookhaven National Lab also recommends 100 percent IPA wash to separate the H_2SO_4

from other sulfates. If, however, the intent is to remove H_2SO_4 and other water soluble material, then Method 5F as proposed will accomplish this separation.

Response: We do not believe that there is any need to operate the sample probe at a temperature higher than that specified in Methods 5B and 5F. Our own tests did show that a higher probe temperature of 205°C (400°F) resulted in the collection of smaller amounts of condensed H_2SO_4 in the probe wash, but it did not completely eliminate it. The most probable explanation for this is that the probe and connecting glassware were not heated uniformly from nozzle to filter, and cold spots in the probe caused H_2SO_4 condensation to occur. Higher temperatures may help reduce the cold spots through conduction, but we have no conclusive data to show that any temperature would guarantee elimination of cold spots and insure that no H_2SO_4 condensed in the sampling system. Therefore, subsequent laboratory treatment to remove any residual H_2SO_4 would be required even if higher probe temperatures were used. As a result, there is no advantage to operating the sample probe at higher temperatures and there may be significant disadvantages because operating at higher temperatures is a much more difficult task.

We agree that 100 percent IPA will dissolve H_2SO_4 without dissolving other sulfates. However, the more important question is whether 100 percent IPA is capable of quantitatively extracting H_2SO_4 from particulate matter samples. Our own results indicate that it is not always effective, and the water extraction is more reliable. Although the water extraction will remove all water soluble sulfates, we believe that H_2SO_4 is the only source of water soluble sulfates at those facilities for which we have proposed its use.

8.3 Comment: It is clear from this proposal that EPA wants to use an ion chromatograph to analyze the sulfate portion of the particulate matter extract. I do not object to the use of the ion chromatograph as the analytical method of choice, but I do object to the exclusion of the barium-thorin titration method as an equally acceptable analytical method.

Response: At the time of the proposal, we did not have data to show that the barium-thorin titration procedure was an acceptable alternative method of measuring sulfate in the water extract. We recently completed the necessary development work and will propose this as an alternative method. This proposal will have to be published as a separate document to allow public comment. The current schedule calls for its publication in 1986.

8.4 Comment: I question the need to extract water soluble sulfate from a soiled filter for 6 to 8 hours using a reflux apparatus (at an unspecified temperature) in order to insure complete extraction. We normally extract water soluble sulfates from a soiled filter using distilled water at room temperature with repeated stirring over a 30-minute period and found this procedure to be acceptable. Does EPA have data to support the need for such a lengthy extraction using the more sophisticated reflux apparatus to achieve complete sulfate extraction? If not, this requirement should be eased.

Response: We do have data to show that the reflux step will quantitatively recover water soluble sulfates from glass fiber filters, but we do not have any comparable information to show that other, less rigorous, extraction procedures will also quantitatively recover the water soluble sulfates. In the absence of such information it would be premature to revise the extraction procedure.

8.5 Comment: It would also appear that only a distilled water blank is being used to correct the particulate matter weight. A distilled water blank and a mass filter blank treated in the same manner as the sample is necessary if EPA decides to use Section 4.3.3 as written. The same is true for the sulfate analysis in 4.3.2.

Response: We agree that insufficient sample blanks are included in Method 5F and have revised the method to require the extraction of a filter blank with subsequent analysis of the extract for sulfate content.

8.6 Comment: Because the weight of a 600-ml beaker is typically about 200 g, the combined weight of the beaker and sample residue will exceed the capacity of most balances that can weigh to 0.1 mg. The EPA needs to choose a beaker whose weight is more in line with the amount of residue left after the evaporation of the water and whose weight will not exceed the capacity of the balance.

Response: We agree that the tare weight of a 600-ml beaker is too high and have amended Method 5F to require a 250-ml beaker instead.

Commenter IV-D-9

9.1 Comment: We agree with EPA that condensed SO_3 should not be included as part of particulate emission measurements, given that use of the best demonstrated technology does not effectively reduce condensed SO_3. Our review of the information in the docket indicates that Method 5B can reduce the measurement of condensed SO_3 at FGD and non-FGD sources. It is puzzling that the agency has limited the application of Method 5B to only those NSPS facilities that are equipped with wet FGD systems. We recommend that the use of Method 5B be permitted for particulate emission testing at all fossil fuel-fired combustion sources, not just those equipped with a wet FGD system.

Response: Method 5B was proposed as a specific solution to a specific problem. The problem is the increased potential for H_2SO_4 emissions as a direct result of the use of higher sulfur content fuels which was made possible by the advent of FGD units. Thus, Method 5B is appropriate for use only at those sources which have this increased potential for H_2SO_4 emissions.

9.2 Comment: The Agency has acknowledged, and the courts have required, that the techniques used by EPA to collect data to establish a standard, and the requirements prescribed for determining compliance with the standard have a known and consistent relationship. We believe that EPA has not demonstrated that Method 5B used after wet FGD systems produces data that are consistent with Method 5 measurements upstream of wet FGD systems. In the absence of such comparative data, we recommend that Subpart Da operators should retain the option of conducting particulate emission tests with Method 5 or 5B, upstream of wet FGD systems.

Response: See Response to Comment 5.1.

LIST OF COMMENTERS

Docket Number A-81-05

Docket Item Number	Commenter/Affiliation
IV-D-1	V.W. Redin, Vice President Plant Operations and Engineering New York State Electric Gas Corporation 4500 Vestal Parkway East Binghamton, New York 13903
IV-D-2	James K. Hambright, Director Bureau of Air Quality Control Department of Environmental Resources Commonwealth of Pennsylvania Post Office Box 2063 Harrisburg, Pennsylvania 17120
IV-D-3	U.V. Henderson, Associate Director Environmental Affairs Research Environment and Safety Department Texaco, Inc. Post Office Box 509 Beacon, New York 12508
IV-D-4	Richard W. Rice, Manager Engineering Sciences Phelps Dodge Corporation 2600 North Central Avenue Phoenix, Arizona 85004
IV-D-5	Peter C. Cunningham Hopping, Boyd, Green and Sams for Florida Electric Power Coordinating Group Post Office Box 6526 Tallahassee, Florida 32314
IV-D-6	Phillip L. Youngblood, Director Air and Toxic Programs Conoco, Inc. Post Office Box 2197 Houston, Texas 77252

LIST OF COMMENTERS
(Continued)

Docket Number A-81-05

Docket Item Number	Commenter/Affiliation
IV-D-7	David R. Pathe, Senior Analyst, Air International Paper Company 77 West 45th Street New York, New York 10036
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IV-D-9	Charles Knaus Hunton and Williams for UARG Post Office Box 19230 Washington, D.C. 20036

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